

Bibliographic Data :

Title : Method for producing high performance active carbon from nut shell

**Application
Number :** 97118398

Application Date : 1997.10.29

**Publication
Number :** 1215694

Publication Date : 1999.05.05

IPC : C01B31/08

Applicant : Hu Fuchang

Inventor : [MT] Hu Fuchang

**Priority
Information :**

A process of preparing high-performance activated carbon with fruit stone or shell includes wetting and puffing the granular fruit stone or shell having size not greater than 30 mm with 50-85% solution of phosphoric acid at 25-130

Abstract : deg.C, charring at 160-250 deg.C, activating at 400-600 deg.C, recovering phosphoric acid, washing in water, drying, crushing and screening. The maximum porous volume is up to 1.6 ml/g and the greatest surface area is up to 2500 sq.m/g. The ratio of middle pores is 40-60%.

Claims

1. Fruit nucleocapsid medicines activate the method (chemical method) to make amorphous granular carbon, raw materials are impregnated with chemicals solution (activator), then the carbomorphism and activation at certain temperature, the characteristic of the invention lies in: Coarse-grained raw materials impregnate and stir and fully totally absorb solution phosphate swellly to raw materials granule at the same time at 25-130 °C with dense solution phosphate, the supplies are loose in shape, then the carbomorphism at 160-250 °C, activate at 400-600 °C.
2. According to the method of claim 1, the characteristic of the invention lies in: The graininess of raw materials is 1-30mm.
3. According to the method of claim 1, the characteristic of the invention lies in: The weight concentration of solution phosphate is 50-85%.
4. According to the method of claim 1, the characteristic of the invention lies in: Carbomorphism and time that time i.e. all supplies keep this temperature after reaching the specified temperature of activation, the hour of carbomorphism 1-3, activate 0.5-2 hours.

-- 1 --

The fruit nucleocapsid makes high-performance activated carbon method

The invention is a method regarding take fruit nucleocapsid as raw materials and make the amorphous granular carbon, one high pore volume, large surface area, mesopore development but consumption of raw materials very low amorphous manufacturing approach of granular carbon.

Produce the fruit nucleocapsid activated carbon method currently, usually put raw materials into carbonization furnace and dry distil carbonomorphism at 300-700 °C first, then pulverize and screen the material of carbonomorphism, open steam, combustion air or the two mixed gas pay activating under 800-1000 °C temperature in the activating oven, call it the gas activation (physical method). The cardinal disadvantage of the this method, first, the product takes capillary as the core, the mesopore is undeveloped, meet great molecular absorption and recovery of solvent of liquid phase, second, the very high of the consumption of raw materials very much, each ton activates charcoal to need to consume fruit more than ten tons of nucleocapsid, even is up to two, 30 tons, the wasteful use of resources is serious. Method (chemical method) to adopt activatory chemicals such as zinc butter, phosphoric acid, etc., can make the hole voluminously with the high extraction rate, the developed activated carbon of mesopore, but this method has been always only used for taking wood dust as raw materials and making activated powdered carbon. The this method demands to activate medicine and fully permeate through the cell tissue of raw materials, because the fruit nucleocapsid is hard to density, the routine method is difficult to achieve the goal. The ancient bronze mirror, the patent application 93120298 of China has proposed pulverizing the fruit nucleocapsid to 20-40 mesh first, then solution phosphate taking concentration as 20-50% is impregnated at 60-100 °C of temperature, flotation method, it is Fructus a raw materials nucleocapsid wood dust substantially. The weak point one of this scheme pulverizes the fruit nucleocapsid weakly, the power consumption is great, and unavailability of fluorescence producing quite a lot of. This scheme weak point two is not high because of the solution strength of phosphoric acid used, even totally soak raw materials, material acid ratio is difficult to improve, can't produce high pore volume, large surface area, mesopore developed product, and because solution phosphate of medium-sized concentration is strong corrosive, when industrialization is implemented, a lot of trouble appears in metropolis on craft and apparatus. This scheme no. The place of the foot can not make the large activated carbon product of graininess three times,

-- 1 --

The finished graininess must be below the graininess of raw materials, so the maximum particle size of product of this scheme should be below 20 mesh (0.9mm).

It is developed to it is an object of the present invention to provide and make high pore volume, large surface area, mesopore, and graininess can in broad range type selective fruit nucleocapsid amorphous a kind of high extraction rate of granular carbon, low-cost and apt operating method.

This inventor is discovered thoroughly, the fruit nucleocapsid is mixed and stirred in definite proportions with high-concentration solution phosphate, then process at certain temperature, can make raw materials swell, puff, achieve the goal that activator permeate evenly, further raise the temperature and finish the plasticizing process and dehydrate carhomorphism, thus has solved the key issue of the invention. The material of carhomorphism is activated at 400-600 °C, has produced the high-quality activated carbon product met purpose requirement of the invention.

Particularly not exceed graininess 30 millimetric air dried fruit nucleocapsids (percentage of moisture let's should higher than 20%) and according to 1:0 of solid-liquid ratios in the phosphate solutions of 50-85% of concentrations (weight), add into processing unit sequentially by 6-1.5, stir and process at 25-130 °C, make acid liquor permeate in the cell tissue of the fruit nucleocapsid gradually, cellulose, hemicellulose and lignin are melted and degraded, the granule is swell, is puffing constantly, free acid liquor is being put and blotted. Wait for the surface of particle to become non-obvious to moisten sense and treachiness, each granule is when being loose in shape, the temperature is improved until 160-250 °C, treatment 1-3hr, because strong water deprivation and catalysis of polyphosphoric acid, the supplies undergo and plasticity reacting then carhomorphism first. The process of this carhomorphism, influence pore structure and absorption performance of the product notably, should make into the developed activated carbon of mesopore, this stage is very necessary. Should master according to raw materials situation. Better condition is 180-220 °C of temperature, time 1-2hr, and stir the supplies to make the material of carhomorphism present the good loose state so as not to bind regularly. Activate the temperature control (the best 450-500 °C) at 400-600 °C, activate time to master flexibly according to raw materials and raw materials amount, generally the material keeps 0.5-2hr of this temperature after reaching the specified temperature warmly, (best 40-60 minutes). Usually activation temperature helps to improve intensity and density of the product high, but the too high temperature is disadvantageous that the mesopore develop. Activate time and pass short mesicin

-- 2 --

Metropolis reduces intensity and absorption performance of the product, activating time will have certain influence on the extraction rate of the product.

The activated material reclaiming phosphoric acid, water washing, dries, pulverizes and sieves according to often method, can get the finished products meeting the requirements.

It can be used for the apparatus pattern of the above-mentioned manufacturing approach, it is not the prototype equipment to adopt existing fixed form in line with the local conditions, but preferably specially design the stirring immersion material apparatus, stirring carbonization furnace and moving bed activating oven.

The strong corrosiveness to the equipment and materials is always difficult point that the chemical method has produced the activated carbon to activate medicines, because the invention adopt triphasic to part homework, prevent anti-corrosion from well as well as fire-resistant contradiction, on the other hand, high-concentration corrosiveness of phosphoric acid minor, condensation polymerization become polyphosphoric acid, diposphoric acid, their little corrosiveness while the charcoal activates, and supplies get wrap protective membrane have good corrosion resistance nature in apparatus surface plasty, thus make charcoal activate choice of equipment and materials comparatively easy, can reduce equipment investment or lengthen life cycle accordingly.

The invention has the following prominent advantages:

First properties of product fine, high pore volume (most large to reach 1.6ml/g), large surface area (most large to up to 2500m²/g). The mesopore is developed (about 40-60% of the whole pore volume), so are very suitable for great molecular absorption and organic recovery of solvent of the gas phase of liquid phase, activate the unsetting particulate charcoal of proper excise to improve by 50-100% in the liquid phase e.g., to the feed liquid best quality physical method of performance ratio of decolorization of industrial citric acid. It is 1-2 times that of above-mentioned physical charcoal in gas phase such as absorption capacity to the fuel gasoline steam, and have effects of desorbing at the good low temperature. Suitable for the fuel of gasoline car to evaporate and use in the controlling device.

The second is that the consumption of raw materials is low, each ton activates charcoal it nearly takes 2-3 tons of fruit nucleocapsid, 10-30% that the material dawdled while only producing for the physical method.

The third is the majority Fructus nucleocapsid does not need and can be used directly through pulverizing, such as the Cortex ziziphi mauritaniae nucleocapsid.

-- 3 --

Apricot nucleocapsid, Cortex Sapi Radicis seed shell, walnut shell, Melia azedarach L. seed shell, etc., cocoon shell, olive nuclear too so long as crushing greater than 30mm to use to graininess, thus has simplified production process, the power saving and consumption of raw materials prominently.

The fourth graininess that is the product can be selected and adjusted and controlled, especially help to make large granulometric activated carbon (maximum particle diameter 20mm) in the broad range type.

The fifth saves the processes and apparatuses of separating raw materials and solution phosphate, and simplified and operated.

The sixth because adopt high-concentration phosphoric acid and triphasic and differentiate the homework, reduce the selective difficulty of equipment and materials.

Further prove with the example as follows

Example 1

Commercially available Cortex ziziphi mauritaniae nucleocapsid 300g (air-seasoned, about 13% of percentage of moisture, particle size about 4-8mm) not pulverized, put in porcelain dish, add solution 300ml 85% phosphate, mix thoroughly, lefts standstill for 17hr at room temperature, then send into accuse of among thermal electric stove automatically, process 2hr at 110 °C, every 0.5hr stirs the supplies once, the carbonomorphism 1.5hr then at 220 °C, activate 40 minutes at 480 °C, take out and cool, reclaim the after washing of phosphoric acid and concocot straight PH6-7, dry at 120 °C, get activation charcoal 116g similar to the raw materials appearance, through pulverizing getting 8-16 mesh amorphous granular carbon 80g, No. 0.29, bulk density, said product / ml, 65% intensity, iodide absorptive value 1001mg/g, methylene blue decolorize strength 13ml, 8 burned sugar decolorize force > 100%, tetrachloro-methane absorption rate 140%, # is 52% of vaporized absorption rate (52.2 °C) for 90 gasoline, desorpt remaining rate (30.2 °C) 12%.

Example 2

Goods apricot nucleocapsid 300g (air-seasoned, about 13% of percentage of moisture, particle size 4-25mm) not pulverized, put in porcelain dish, add solution 300ml 85% phosphate, mixes thoroughly, lefts standstill for 18hr at room temperature, then send into and accuse of in the thermal electric stove automatically, 120 °C treatment 3hr, stirs the supplies once every 0.5hr, the carbonomorphism 1.5hr then at 220 °C, 480 °C activates 40 minutes, take out

-- 4 --

Cool, reclaim the after washing of phosphoric acid and concoc straight PH6-7, oven dry at 120 °C, gets activation charcoal 102g similar to the raw materials appearance, through pulverizing 8-16 mesh amorphous granular carbon 70g. Said product bulk density 0.32g/ml, intensity 72%, iodide absorptive value 950mg/g, methylene blue to decolorize strength 12ml, 8 burned sugar decolorize force > 100%, 120%, acetic acid of absorption rate of tetrachloro-methane absorbs (24-48 mesh) 720mg/g, # 49% of vaporized absorption rate (52.2 °C) for 90 gasoline, desorpt remaining rate (30.2 °C) 13%.

Example 3

Cortex Sapiti Radicis seed shell 300g (about 11% of percentage of moisture, particle size 2-7mm), put in porcelain dish, add and reclaim dense phosphoric acid (concentration is about 85%) 300ml, mix thoroughly, send into accuse of among thermal electric stove automatically, 110 °C treatment 3hr, every 0.5hr stirs the supplies once, the carbonomorphism 2hr then at 200 °C, 480 °C activates 40 minutes, take out and cool, reclaim the after washing of phosphoric acid and concoc straight PH6-7, oven dry at 120 °C, get activation charcoal 140g similar to the raw materials appearance to get 24-48 mesh amorphous granular carbon 108g after pulverizing, said product bulk density 0.36g/ml, intensity is 90%, iodide absorptive value 980mg/g, methylene blue decolorize strength 12.5ml, tetrachloro-methane absorption rate 115%, acetic acid absorbs (24-48 mesh) 660mg/g, # 90 gasoline vaporized absorption rate (52.2 °C) 48g, desorpts 14% of remaining rate.

Example 4

Walnut shell 120g (air-seasoned, aqueous about 14% particle size about 5-15mm), puts in porcelain dish, adds solution 100ml 85% phosphate, mix thoroughly, send into accuse of among thermal electric stove automatically, 120 °C treatment 3hr, every 0.5hr stirs the supplies once, 500 °C activates 40 minutes, take out and cool, reclaim the after washing of phosphoric acid and concoc straight PH6-7, oven dry at 120 °C, must be similar to activation charcoal 48g of the raw materials appearance, get 8-16 mesh amorphous granular carbon 32g after pulverizing. Said product bulk density 0.28g/ml, iodide absorptive value 900mg/g, methylene blue decolorizes 40% of vaporized absorption rate (25.2 °C) for 90 gasoline strength 11ml, #, desorpt remaining rate (25.2 °C) 11.5%.

-- 5 --